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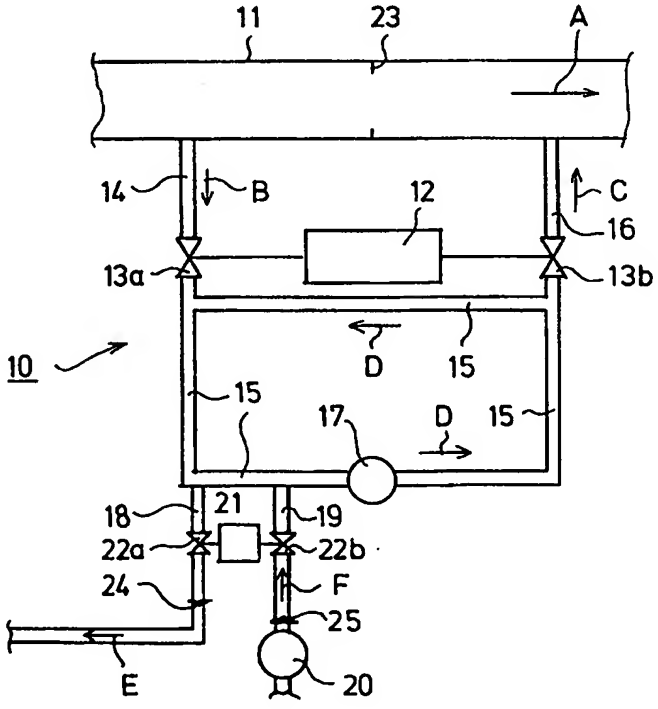
(57) Abstract

The present invention relates to an automatic sampling and treatment system (10) comprising a sampling loop (15) connected on–line to a process (11) to receive a sample stream from a stream (A) being processed in the process (11). The sampling loop (15) is further connected to an analyser to convey the sample stream from the sampling loop (15) to the analyser. The sampling loop (15) is connected to the process (11) with a first conduit (14), and with a second conduit (16). The sampling loop (15) is connected to the analyser with a third conduit (18), and to a feeding system of an additive with a fourth conduit (19). A first actuator (12) is adapted to open and close a valve (13a) in the first conduit (14), and a valve (13b) in the second conduit (16). A second actuator (21) is adapted to open and close a valve (2a) in the third conduit (18), and a valve (22b) in the fourth conduit (19).



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Automatic sampling and treatment system

The present invention relates to an automatic sampling and treatment system comprising a sampling loop connected on-line to a process to receive a sample stream from a stream being processed in the process, and further, connected to an analyser to convey the sample stream from the sampling loop to the analyser.

Continuously operating analysers are used in the chemical industry for quality control to determine concentrations or other important quality criteria from the process streams desired. One of the most important features of these on-line analysers influencing the performance thereof is a sampling system that functions properly.

In the petroleum refining, dark and heavy, high boiling crude oil fractions and mixtures thereof are formed, and thereafter, used as starting materials to produce bitumen and heavy fuel oil. The poor solubility or the precipitation of asphaltenes contained in these refined oil products impedes the application and storage thereof. The tendency of the asphaltenes to precipitate determines the stability, or the storability of these oil products, this tendency depending on the process, and the starting materials used in the production. Particularly the thermal cracking used at present influences the tendency of the asphaltenes to precipitate. In the thermal cracking unit, the crude oil refining process is controlled in such a way that the heavy fuel oil obtained from the bottom of the unit were stable.

Traditionally, the stability of oil products is determined from samples taken with the so-called flocculation method, among others (for instance, the Shell method; Flocculation ratio of bitumen and fuel oil). The method is based on the paper chromatography, and the visual evaluation of the chromatograms. With this method it takes 0,5-6 hours to obtain the results.

Automatic on-line analysers have also been developed, the function thereof being for instance the monitoring of the asphaltene content of the oil product, either directly from the production process, or by analysing the samples in a laboratory.

In case of an automatic on-line analyser, a process analyser controlled by a computer automatically takes a sample from the product stream desired, produces a parameter characterising the stabilility, and sends it to the control unit of the process at predetermined intervals, for instance every 10 minutes.

The object of the present invention is to improve the systems known at present.

The automatic sampling and treatment system of the invention is characterized in that the sampling loop is connected on-line to the process with a first conduit, and with a second conduit, the sampling loop is connected to the analyser with a third conduit, and to a feeding system of an additive with a fourth conduit, and that a first actuator is adapted to open and close a valve in the first conduit, and a valve in the second conduit.

In a preferable embodiment of the present invention a second actuator is adapted to open and close a valve in a third conduit, and a valve in a fourth conduit. The valve actuated by the second actuator is not necessarily needed in the third conduit, but can be replaced with a return valve. The second actuator is not necessary, since it may be replaced with a pump, if desired, allowing also the valves in the third and fourth conduits to be omitted, if desired.

The functioning of the sampling and treatment system according to the invention, used as a part of a continuously operating analyser, is based on the constant volume sampling known as such, the said system also allowing, however, the treatment of the sample for analysis. The sampling and treatment system of the invention takes from a process of a production plant a liquid sample with a predetermined volume, and carries out the treatment, such as the agitation and the precipitation of the sample taken.

According to a preferable embodiment of the invention a precipitating solvent is continuously added to a closed liquid circuit, and accordingly, a respective amount of liquid is removed from this closed circuit, this liquid flowing to the detector that detects the precipitation as the sudden increase (the turning point) of the intensity of the scattered (reflected) light. The parameter (p value, stability) illustrating the tendency to precipitate is calculated from the quantities of the solvents and oil products.

An oil sample with a constant volume is necessary for the calculation of the parameter illustrating the tendency to precipitate, determined by the relative amounts of the oil, the precipitating solvent (a straight-chain hydrocarbon, such as a heptane, pentane, cetane, octane, etc.), and possibly a solvent (xylene, toluene, or another aromatic compound).

The sampling and treatment system of the invention is advantageous for the analysis of liquids that are difficult to pump. In addition to homogeneous liquids, the liquid sample may also be a so-called inhomogeneous sol with a high viscosity, or a liquid containing solid particles (a slurry), the invention being, however, applicable to any liquid sample.

In the system according to the present invention the mixing during the treatment of the sample is almost ideal, accomplished for instance with a (gear) pump. In the precipitation step, the treatment of the liquid is characterized by the continuous feeding of the precipitating agent (such as heptane) into the mixing loop with a constant volume, as well as by the flow of the liquid volume being displaced through the detector (i.e. the detection of the degree of precipitation). The mixing loop may be equipped with elements that make the mixing more efficient, for instance with a (gear) pump, static mixers, packings, beads etc.

The equation for the degree of precipitation is:

$$Q_t = Q_0 (1 - e^{-t F_h/V_0})$$

where

Q_t = concentration of the precipitating agent (e.g. heptane)

Q_0 = final concentration

t = time

V_0 = volume of the sample loop

F_h = flow of the precipitating agent (e.g. heptane)

The above equation may for instance be used for the precice determination of the concentration of the liquid in the sample loop when the concentration measured by the detector is known.

The automatic sampling and treatment system of the present invention is accompanied with several significant advantages. The sampling, and the treatment of the sample are accomplished in a closed space which is particularly important when handling inflammable or toxic substances. The treated liquid is automatically removed from the sample loop, and recycled to the process during the next sampling step, thus reducing the amount of waste and the need to wash the loop. The sampling, and the treatment of the sample are not influenced by the pressure of the process, or pressure variations therein. The sampling, and the treatment of the sample may be automatized easily. The flow to the analyser may be precisely adjusted within a wide range. The reagent used is consumed only in amounts necessary for the treatment.

The sampling and treatment system of the present invention is especially suitable for the automatic sampling from the process flow of heavy fuel oil, for an on-line analyser. The sampling and treatment system of the present invention may of course be used for the sampling of any liquid from any process flow.

The invention is now illustrated with reference to the figure of the appended drawing, showing the principle of the invention, naturally without intending to limit the invention exclusively thereto.

The figure of the drawing shows a preferable embodiment of the sampling and treatment system of the invention as a schematic flow chart.

In the figure of the drawing, the automatic sampling and treatment system of the invention is generally referred to with the numeral 10. The liquid being processed flows in the direction of the arrow A in the process 11. The automatic sampling and treatment system 10 is connected with a first conduit 14, and with a second conduit 16 to the process 11. The conduit 14 is adapted with a valve 13a, and the conduit 16 is adapted with a valve 13b. Preferably, the valves 13a, 13b form a pair of two-way valves that are actuated coincidently. An actuator 12 is adapted to control the valves 13a, 13b. Further, the sampling and treatment system 10 comprises a sampling loop 15. The sample stream flows in the direction of the arrow B from the process 11 through the conduit 14 into the sampling loop 15. The sampling loop 15 is preferably equipped with a pump 17. In the sampling loop 15 the sample flows in the direction of the arrow D. In the next samplig step, the treated liquid is automatically removed from the sampling loop 15 through the conduit 16 as the stream C back into the process 11.

The sample stream leaves the sampling loop15, through a conduit 18 as the stream E towards the analyser. An additive is fed through a conduit 19 into the sampling loop 15. In this embodiment, the additive is fed with a pump 20. The conduit 18 comprises a valve 22a, and the conduit 19 comprises a valve 22b. An actuator 21 is adapted to control the valves 22a,22b. Preferably the valves 22a,22b form a pair of two-way valves that are actuated coincidently. The additive flow through the conduit 19 is illustrated with the arrow F.

Preferably, in the embodiment shown in this figure the process 11 comprises a flow resistance, in this case a throttling baffle 23. The conduit 18 leading to the analyser

comprises a pressure reducer 24 to prevent excessive liquid from leaking from the sampling loop 15, thus assuring the reliable performance of the sampling and treatment system of the invention. The conduit 19 to convey the additive comprises a return valve 25.

The operation of the automatic sampling and treatment system 10 according to the invention is as follows. During sampling, the actuator 12 first opens the sampling loop 15, the liquid from the process stream desired thus displacing the liquid already present in the sampling loop 15. This is facilitated by a pressure difference caused by restricting the flow in the process 11 with a flow resistance, e.g. with a throttling baffle 23, or a separate pump in the sample line (not shown). Also the pump 17 assists in filling the sample loop 15.

At the beginning of the sample treatment, the actuator 12 closes the valves 13a, 13b, that is, it closes the connection between the sampling loop 15, and the process 11. Then, the actuator 21 opens the valves 22a,22b, thus allowing the additive to flow into the sample loop 15 through the conduit 19. During the sampling and the sample treatment, the sample is effectively circulated by the pump 17 in the sampling loop 15. The additive is fed by the pump 20 into the sampling loop 15 where the mixing is almost ideal. The additive feed displaces a respective amount of liquid that now flows towards the analyser through the conduit 18. The feeding of this precipitating additive is continued until the parameter characterizing the tendency to precipitate is reached. Thereafter, the actuator 21 closes the valves 22a, 22b, and the actuator 12 opens the valves 13a,13b to allow the sampling loop 15 to receive a new sample. The treated liquid in the sampling loop 15 is automatically removed through the conduit 16 and recycled to the process during the next sampling step, thus reducing the amount of waste being formed and the need to rinse the loop. The flow to the analyser may be precisely adjusted within a wide range with the aid of the pump 20. The reagent fed with the pump 20 is consumed only in amounts necessary for the treatment.

Only the principle of the present invention is presented above, and it is evident to the person skilled in the art that several modifications are possible within the spirit and scope of the invention defined in the appended claims.

Claims

1. Automatic sampling and treatment system (10), comprising a sampling loop (15) connected on-line to a process (11) to receive a sample stream from a stream (A) being processed in the process (11), and further connected to an analyser to convey the sample stream from the sampling loop (15) to the analyser, **characterized** in that the sampling loop (15) is connected on-line to the process (11) with a first conduit (14), and with a second conduit (16), the sampling loop (15) is connected to an analyser with a third conduit (18), and to a feeding system of an additive with a fourth conduit (19), and that a first actuator (12) is adapted to open and close a valve (13a) in the first conduit (14), and a valve (13b) in the second conduit (16).

2. The automatic sampling and treatment system according to the claim 1, **characterized** in that a second actuator (21) is adapted to open and close a valve (22a) in a third conduit (18), and a valve (22b) in a fourth conduit (19).

3. The automatic sampling and treatment system according to the claim 1, **characterized** in that the valve in the third conduit is a return valve.

4. The automatic sampling and treatment system according to any of the above claims 1—3, **characterized** in that a pump (20) is adapted to feed an additive through the fourth conduit (19) into the sampling loop (15).

5. The automatic sampling and treatment system according to the claim 4, **characterized** in that the additive comprises a precipitating additive.

6. The automatic sampling and treatment system according to the claim 5, **characterized** in that the precipitating additive comprises a straight-chain hydrocarbon, preferably a heptane, pentane, cetane, or octane.

7. The automatic sampling and treatment system according to any of the above claims 1—6, **characterized** in that the sampling loop (15) is equipped with a pump

(17) adapted to circulate the sample stream, and to support thorough mixing during the sampling, and the sample treatment.

8. The automatic sampling and treatment system according to any of the above claims 1—7, **characterized** in that the treated sample stream is adapted to leave the sampling loop (15) through the second conduit (16) towards the process (11) during the next sampling step.

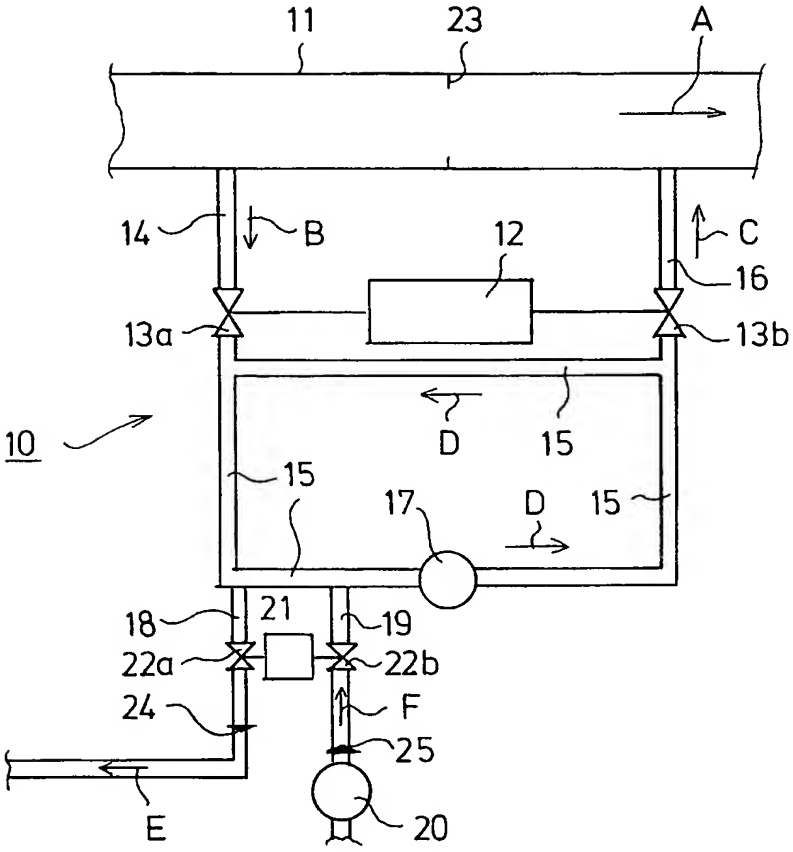
9. The automatic sampling and treatment system according to any of the above claims 1—8, **characterized** in that the process (11) comprises a flow resistance.

10. The automatic sampling and treatment system according to the claim 9, **characterized** in that the flow resistance is a throttling baffle (23).

11. The automatic sampling and treatment system according to any of the above claims 1—10, **characterized** in that the third conduit (18) leading to the analyser is equipped with a pressure reducer (24).

12. The automatic sampling and treatment system according to any of the above claims 1—11, **characterized** in that the conduit (19) for feeding the additive is equipped with a return valve (25).

13. The automatic sampling and treatment system according to any of the above claims 1—12, **characterized** in that the valves (13a,13b) in the first and second conduits (14,16), as well as the valves (22a, 22b) in the third and fourth conduits (18, 19) are respectively two-way valves.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INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 98/00519

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: G01N 1/10, G01N 1/20
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 9114932 A1 (MüTEK GMBH), 3 October 1991 (03.10.91), page 4, line 28 - page 6, line 20	1,5
Y		1
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Y	GB 2170909 A (SPECTRA-TEK UK LIMITED), 13 August 1986 (13.08.86), page 1, line 54 - page 2, line 9; page 2, line 52 - line 63	1
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A	GB 2206408 A (NIGEL DAVID EVANS), 5 January 1989 (05.01.89), page 4, line 12 - line 24	1-13
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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5275786 A (DONALD D. SOLETA ET AL), 4 January 1994 (04.01.94), column 3, line 30 - line 68 --	1-13
A	GB 2164021 A (JISKOOT AUTOCONTROL LIMITED), 12 March 1986 (12.03.86), page 1, line 32 - line 55 -- --------	1-13

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Information on patent family members

27/07/98

International application No.
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